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Tribology of Selected Ceramics at Temperatures to 900 °C

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TRIBOLOGY OF SELECTED CERAMICS AT TEMPERATURES TO 900 °C

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SUMMARY

Results of fundamental and focused research on the tribological properties of ceramics are discussed. The basic friction and wear characteristics are given for ceramics of interest for use in gas turbine, adiabatic diesel, and Stirling engine applications. The importance of metal oxides in ceramic/metal sliding combinations is illustrated. The formulation and tribological evaluation of composite, plasma sprayed ceramics with solid lubricant additives are described. Friction and wear data are given for carbide and oxide-based composite coatings for temperatures to at least 900 °C.

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INTRODUCTION

As ceramics become more frequently used in engine components, it is clear that their friction and wear properties must be better understood. It is also clear that surface modifications or other means of providing acceptable levels of friction and wear will be needed because ceramics, for the most part, do not have inherently good tribological properties. Friction coefficients in excess of 0.7 have been reported for example by Sutor,[1] and Sikra et al.[2] for silicon nitride sliding on itself or on M50 bearing steel. Breznik et al.[3] reported on the friction and wear behavior of like and unlike combinations of sintered alpha SiC, siliconized SiC, and a yttrium stabilized zirconia. In every case they observed high steady state friction accompanied by considerable wear. Cranmer reported similar results for monolithic, silicon-based ceramics.[4] All of these studies confirm that ceramic tribological components need to be lubricated. This is a significant challenge because application temperatures are often higher than the thermal oxidation limits of lubricating

oils and indeed of conventional solid lubricants such as graphite and molybdenum disulfide. Therefore, research to develop high temperature solid lubricants and lubrication methods for ceramics is a logical approach to this problem. Lubrication is important, not only for ceramic/ceramic contacts, but also for ceramic/metal contacts because some critical bearing and seal applications involve ceramics in sliding[5,6] or rolling[7] contact with metal surfaces.

In this study, we determined the friction and wear of selected ceramics sliding on a precipitation hardened nickel base super alloy at room temperature and at 800 °C in order to study the possible lubricating effects of metal oxidation on the tribological properties of ceramic/metal sliding combinations. A more detailed parametric study was then performed on the effects of temperature and sliding velocity on friction and surface characteristics to 900 °C.

Plasma sprayed zirconia was modified by the addition of calcium fluoride in an attempt to improve its tribological properties. Finally, a composite, plasma sprayed coating composition was formulated consisting of metal-bonded chromium carbide for wear resistance, and solid lubricant additives. The solid lubricant additives were metallic silver and a eutectic of barium fluoride and calcium fluoride. The friction and wear characteristics of this composite coating were evaluated from 25 to 760 °C in atmospheres of air, helium, and hydrogen.

The objective of the experiments in helium and hydrogen was to identify a candidate piston ring and cylinder liner material combination with acceptable friction and wear properties for use in the hot areas of the Stirling engine cylinders. This was done in support of a Stirling engine program described in Ref. 8.

EXPERIMENTAL PROCEDURE

Friction and wear data were obtained with two different types of wear specimen configuration: a double rub shoe on disk arrangement, Fig. 1(a), and a pin on disk arrangement, Fig. 1(b). Experiments were performed in air with a relative humidity at 25 °C of 50 percent, in dry helium, or dry hydrogen. The duration of most experiments was 1 hr. Friction was measured continuously during the experiments. Wear was measured periodically, usually every 20 min, in order to determine the transition from run in wear to steady state wear. The wear factors given in this paper are for steady state wear.

Wear factor, k,

Wear is expressed in this paper as a wear factor which relates volumetric wear to sliding distance (or duration at a given sliding velocity) and to load. Use of this factor assumes that wear volume is directly proportional to the product of the load and the sliding distance. Although this assumption is an over simplification, it has been found to be a reasonable one for steady state wear after a higher wear rate run in period is completed. Comparison of wear factors then allows one to estimate the relative wear resistance of various sliding combinations. Characteristic wear versus sliding duration behavior is shown schematically in Fig. 2, along with the wear factor equation, and a physical interpretation of wear factors in terms of wear severity. Wear factors varied in magnitude from 10^{-7} to 10^{-11} cm^2/kg, with 10^{-7} indicating unacceptably high wear for any application and 10^{-10} to 10^{-11} indicating the wear rates needed for long life sliding components.

RESULTS AND DISCUSSION

Influence of Oxidation on the Friction and Wear of Ceramics Sliding on a Precipitation-Hardened Nickel-Base Super Alloy (Inconel 718)

A series of experiments was performed employing a double rub shoe tribometer. In these experiments, ceramic rub shoes were in sliding contact with the outer rim of a rotating metal disk that was directly heated with an

induction coil. A schematic of the specimen configuration is given on Fig. 1(a). Sliding velocity was 0.50 m/s, and load was 67 N/shoe. The experiments were performed in air with a room temperature relative humidity of 50 percent.

Friction. The friction coefficients for a variety of ceramics are summarized in Fig. 3. A comparison of the friction data immediately shows that while there is a large spread of friction coefficients for the various ceramics in room temperature (no external heating) experiments, there is little variation at 800 °C. The silicon ceramics: alpha SiC and two Si_3N_4 materials exhibited high friction coefficients of 0.5 to 0.6 at room temperature. The oxide ceramics exhibited somewhat lower friction coefficients of 0.43 for polycrystalline Al_2O_3, 0.38 for mullite. ($3Al_2O_3.2SiO_2$) and 0.3 for quartz. At 800 °C on the other hand, the friction coefficients for all the ceramics were about 0.3, the same as for the nickel alloy sliding against itself. These observations strongly imply that the friction for the ceramic/metal sliding combinations at elevated temperature under oxidizing conditions is predominantly controlled by the frictional characteristics of the oxidized metal. Some interactions can be expected among the oxides on the metal and those on the ceramics, but the very small differences in friction coefficients at 800 °C for all the ceramic/metal combinations and the fact that they are all nearly identical with the metal/metal friction coefficient, strongly supports the conclusion that the oxidized metal surface controls the friction coefficient. Surface analytical studies performed during a more detailed study of SiC sliding on metal will be described later in this paper.

Wear. Wear factors for the various ceramic/metal combinations are compared in Fig. 4. Metal/metal wear is also included for reference. Figure 4(a) compares wear of the rub shoes at 25 and 800 °C. Analagous data for wear of

the metal disks is given in Fig. 4(b). The sum of shoe and disk wear for each experiment is given in Fig. 4(c).

In room temperature experiments, there was no direct correlation between friction and wear. In fact the relationship was almost inverse. Quartz, mullite, and alumina, which had the lowest friction coefficients, exhibited the highest wear, but they in turn tended to be less abrasive to the metal disks. The lowest combined wear factors (ceramic rub shoe plus metal disk) were obtained for alpha SiC followed by mullite. The highest combined wear coefficients were obtained for the nickel alloy sliding against itself and for quartz rub shoes sliding against nickel alloy disks.

At 800 °C, where all ceramic/metal combinations and the metal/metal couple had about the same friction coefficients, large differences in wear were nevertheless observed. Very low wear of ceramic rub shoes were obtained with the silicon nitrides, (particularly MgO-doped Si_3N_4), mullite, and alumina. Somewhat higher wear occurred with quartz, alpha SiC, and nickel alloy rub shoes, but all rub shoe wear factors were less than 10^{-9} cm^2/kg at 800 °C. Wear of the metal disks tended to be higher than rub shoe wear, but not extremely severe, with all wear factors in the range of 10^{-10} to 10^{-9} cm^2/kg. The lowest disk wear was observed, both at 25 and 800 °C when mullite rub shoes were used. Also, the lowest combined rub shoe plus disk wear factors were obtained at 800 °C with mullite. The lowest combined wear factors at room temperature were achieved with alpha SiC, with the next higher values for mullite.

<u>Significant observations for this series of experiments</u>. (1) Wear factors for silicon nitride, especially for MgO-doped Si_3N_4, are exceptionally low but wear of the counterface metal is severe at room temperature; (2) sintered Alpha SiC is not as wear resistant as Y_2O_3 and MgO-doped silicon nitrides at 800 °C, but it is less abrasive to the nickel alloy and actually exhibited

lower combined wear coefficients; (3) Room temperature friction coefficients of SiC and Si_3N_4 are high, about 0.6, compared to about 0.4 for mullite. Therefore, from an overall consideration of both friction and wear at both 25 and 800 °C, the best of the rub shoe materials studied for sliding contact with nickel alloy is mullite.

Influence of Experimental Parameters on the Tribological Behavior of Alpha SiC Sliding on M50 Bearing Steel

Friction and wear. SiC pins with a hemispherical tip of 4.76 mm radius were slid against M50 steel disks at 25 and 300 °C in an air atmosphere. The specimen configuration is given in Fig. 1(b). Load was varied from 1N to 20N, and sliding velocity was varied from 0.4 to 10.7 m/s. The friction results are summarized in Fig. 5. There was a considerable scatter in friction coefficients which obscured all but the most obvious trends. At room temperature, friction coefficients were 0.25 to 0.45. There was no obvious trend with load, but there appears to be a real trend to higher friction with increasing sliding velocity.

At 300 °C, there again was no trend with load, but, in contrast to the trend at room temperature, there was a very marked decrease in the magnitude and the scatter in friction coefficient with increasing sliding velocity.

It is difficult to attempt an explanation of the influence of sliding velocity on friction, especially in the absence of any apparent corollary influence of load. It is conceivable that the room temperature trend is due to the breakdown of absorbed films at the higher sliding velocities. On the other hand, at 300 °C, the combination of higher bulk temperature and increased surface temperature at the higher sliding velocities may result in the formation of beneficial oxide films on the M50 disk. (After the 300 °C tests the disks were lightly oxidized with a more opaque glazed oxide on the wear track than away from it.)

At both temperatures wear of the SiC pins was very low with wear factors at the low end of the 10^{-10} cm^2/kg range. Wear of the M50 disks was an order of magnitude higher (in the moderate wear regime) with wear factors typically of about $2x10^{-9}$ cm^2/kg.

Surface characterization. SEM photographs of a SiC pin after a friction and wear test at 300 °C are given in Fig. 6. Some scattered, loose wear debris is evident, but no significant amount of adherent material that may have been transferred from the M50 is apparent. EDS analyses gave spectra typical of SiC with no extraneous peaks except when the loose particles were in the field of the analyzing beam, in which case iron, probably in the form of iron oxides, was also detected. The SiC wear surface is highly polished. This is especially apparent in Figs. 6(c) and (d). Numerous voids are distributed over the polished wear surface. These voids were probably present in the original material rather than having been plucked out during the wear process because they are much larger than the SiC wear debris particles.

Influence of Experimental Parameters on the Tribological Behavior of Alpha SiC Sliding on Precipitation-Hardened Nickel Alloy (Inconel X-750)

Friction. Experiments in this series were performed at room temperature and at 900 °C in air, Fig. 7. As with SiC/M50, the friction coefficients at room temperature were very erratic with large data scatter, and there was a trend to higher friction with increasing sliding velocity. The friction at any given velocity on the average tended to be higher than it was for SiC/M50. However, at 900 °C, scatter was much less. Friction coefficients were about 0.4 at low sliding velocities, but above about 3 m/s, the friction coefficient was very consistent at a value of 0.2.

Surface characterization. The SEM photographs on Figs. 8 and 9 and EDS analyses gave a clear indication of why friction decreased so dramatically at 900 °C. Much debris, identified by EDS as originating from the metal alloy,

consisted of metal oxides with randomly scattered SiC particles. The significant feature of the debris that is clearly seen on the photomicrographs is that it has undergone severe plastic deformation. It is characteristic of solid lubricant materials that they shear by plastic deformation rather than by brittle fracture within the sliding contact. Materials that exhibit a brittle to ductile transition temperature are nonlubricative below the transition temperature but may be lubricative at higher temperatures. This is the case for materials such as CaF_2, BaF_2, and LiF, and appears to be the case for oxides formed by the oxidation of nickel-chromium alloys.

Wear. Although the nickel alloy is softer (R_c40) than M50 bearing steel (R_c62), wear of SiC against the nickel-base super alloy was about an order of magnitude higher at both temperatures. Wear of the nickel alloy disks was very severe at room temperature with wear factors in the 10^{-7} cm^2/kg range. At 900 °C, where lubricious oxides provided some protection, wear factors were typically at the high end of the 10^{-9} cm^2/kg range.

Influence of Atmosphere on SiC/metal Friction

Figure 10 compares the friction/temperature characteristics of SiC in vacuum[9] and in air (this work). Because of the large scatter in friction coefficient, especially at the lower temperatures, the values given on this figure are only typical, but the trends with temperature are quite reproducible. The important point is that the atmosphere had a profound effect on the friction coefficient at any given temperature. In vacuum, the room temperature friction coefficient of 0.4 rapidly increases to very high values as the temperature is increased and absorbed contaminants are desorbed. At about 800 °C, friction drops sharply and is about 0.4 at 1200 °C. Surface analyses by x-ray photoelectron spectroscopy (XPS) showed that this reduction in friction at elevated temperature occurs because SiC thermally dissociates at the surface to silicon and graphitic carbon. The reduction in friction for

SiC sliding on unoxidized metal in vacuum at temperatures above 800 °C was attributed in Ref. 9 to this graphitic surface film.

In air by comparison, friction remains at room temperature levels until oxidation of the metal counterface provides a lubrication effect. Graphitic carbon is not likely to be a factor here because any free carbon that may occur as an intermediate product when SiC oxidizes to SiO_2 would most likely be rapidly converted to carbon monoxide and/or carbon dioxide above about 500 °C.

Plasma Sprayed Zirconia/Calcium Fluoride Coatings

A series of pin on disk experiments were performed to determine the effect of calcium fluoride (CaF_2) additions on the friction and wear properties of plasma sprayed zirconia (ZrO_2) coatings with a diamond ground surface finish. Hardened nickel alloy pins were used at a load of 5N and a sliding velocity of 2.7 m/s in an air atmosphere. The data are summarized in Fig. 11. The CaF_2 additions resulted in a modest reduction in friction coefficients. The minimum friction was 0.4 with a 10 percent addition of the fluoride. However, there was a continuous increase in coating wear with increasing fluoride content. The increased coating wear may be attributable to a further weakening of the rather porous coating by the calcium fluoride additions making it unable to withstand the ball on flat type of concentrated contact used in these experiments. The fluoride additions were therefore not considered to be beneficial additives for plasma sprayed ZrO_2 in this simple binary system.

A New Composite Self-Lubricating Coating: PS200

An approach was taken to formulate a composite plasma sprayed coating containing chromium carbide for wear resistance, and additive constituents for reduced friction and to minimize abrasive wear of softer counterface materials. Previous research had demonstrated that wide temperature spectrum self-lubricating coatings could be formulated using stable fluorides for high temperature lubrication and silver to control friction at low temperatures;

all in a superalloy matrix.[10] These coatings have friction coefficients of about 0.2 from cryogenic temperatures to at least 900 °C. However, they are only moderately wear resistant-adequate for low speed or short duration applications (k in the 10^{-9} cm^2/kg range), but not for the long life requirements of a commercially feasible heat engine. Therefore chromium carbide based compositions were investigated in an attempt to achieve both low wear and low friction. A number of chromium carbide-based formulation were developed. One of the more successful to date has the chemical composition given in table I. This coating, which we have designated PS200 consists of 80 wt % metal bonded carbide and 10 wt % each of a fluoride eutectic and silver metal. This coating was evaluated as a lubricant to protect foil bearings during starts and stops when surface velocities are inadequate to provide fluid film lubrication. The results were reported in Ref. 11. It was shown that the formulated coating provided lower starting torque and better endurance than baseline metal bonded chromium carbide. Figure 12 shows that the formulation was also successful in minimizing wear of the bearing foils. Bearing temperature was cycled between 25 and 650 °C in these bearing tests. PS200 is also of potential interest as a cylinder liner material for the Stirling engine which uses helium or hydrogen as the thermodynamic working fluid. Pin on disk studies have been performed to determine a suitable piston ring material for use against PS200 cylinder coatings. The best alloy so far evaluated from a tribological point of view is a hardenable cobalt base alloy (Stellite 6B). Friction data for the cobalt alloy/PS200 sliding combination in helium, hydrogen, and air at 25, 350, and 760 °C are given in Fig. 13, and compared with the results obtained in identical experiments with the baseline carbide coating. Friction is clearly lower for the formulated coating under all conditions studied. The lowest friction was obtained in the hydrogen atmosphere, especially at 760 °C where the friction coefficient was 0.15.

Wear factors for the pin and disk were very low (10^{-11} to 10^{-10} cm^2/kg) and in most cases actually lower than obtained with the baseline coating.

CONCLUSIONS

1. None of the ceramics evaluated in unlubricated sliding contact with a precipitation hardened nickel base super alloy had acceptable friction and wear characteristics at room temperature. At 800 °C in air the metal oxides formed on the metal provided a degree of lubrication. Friction coefficients of about 0.3 were observed for all of the ceramic/metal combinations as well as the metal/metal control. In spite of the uniformity of friction for all the combinations at 800 °C, there was considerable variation in wear factors among the various sliding combinations.

2. Over all, the best unlubricated friction and wear behavior against the metal counterface was obtained with mullite. Silicon carbide wear was comparable with mullite, but its friction coefficient was higher at room temperature.

3. The silicon nitride wear was extremely low, but high wear occurred on the metal counterface sliding against the ceramic, and the friction was high at room temperature.

4. Attempts to improve the tribological properties of plasma sprayed zirconia by adding calcium fluoride to the coating composition were unsuccessful. Calcium fluoride additions up to 10 percent reduced friction, but increased coating wear.

5. A composite plasma sprayed coating (PS200), based upon chromium carbide with solid lubricant additions of metallic silver and a barium fluoride/calcium fluoride eutectic was very wear resistant from 25 to 760 °C in air, helium, and hydrogen. Friction coefficients did not exceed 0.25 in hydrogen. These results suggest that this coating may be a good candidate as a cylinder liner material for Stirling engines utilizing a hydrogen working fluid.

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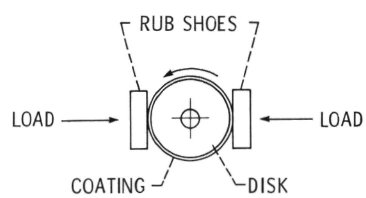
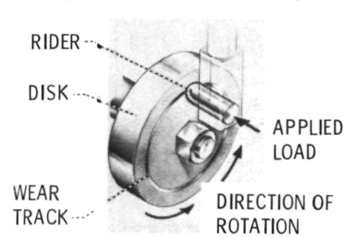
TABLE I

Component	Composition wt %
PS-200	
Cr_3C_2	43
Metal binder	37
BaF_2/CaF_2 eutectic	10
Ag	10
Eutectic	
BaF_2	62
CaF_2	38



(a) Double rub shoe on disk.

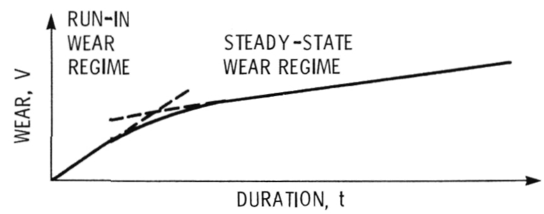


(b) Pin on disk.

Figure 1. - Friction and wear test specimen configurations.

EQUATION: $V = kSW$ OR $V = kvtW$

WHERE:
V = WEAR VOLUME, cm^3
S = SLIDING DISTANCE, cm
W = LOAD, kg
v = VELOCITY, cm/sec
T = TIME, sec



PHYSICAL INTERPRETATION:

$k \leq 10^{-11}$ cm^2/kg (VERY LOW WEAR)

$k \approx 10^{-10}$ TO 10^{-9} cm^2/kg (LOW TO MODERATE WEAR)

$k \geq 10^{-8}$ cm^2/kg (HIGH WEAR)

Figure 2. - Wear factor, k.

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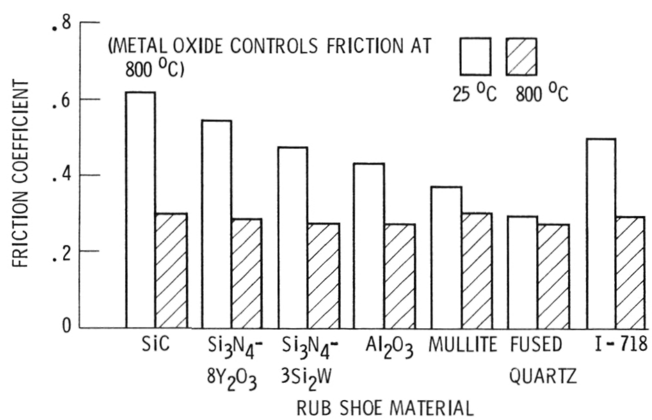

Figure 3. - Friction of various ceramics and of Inconel 718 sliding on Inconel 718 in air (50% R. H.) at room temperature and at 800 °C, 0.18 m/s, 67 N load.

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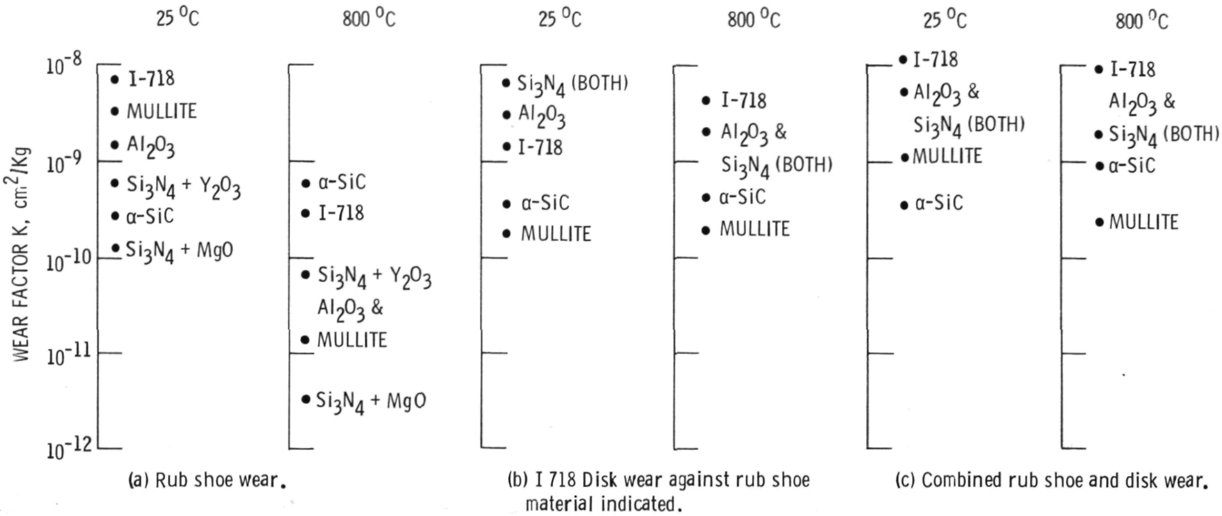

Figure 4. - Wear factors for ceramics sliding on Inconel 718 in air (50% R.H.), 0.18 m/s, 67 N load.

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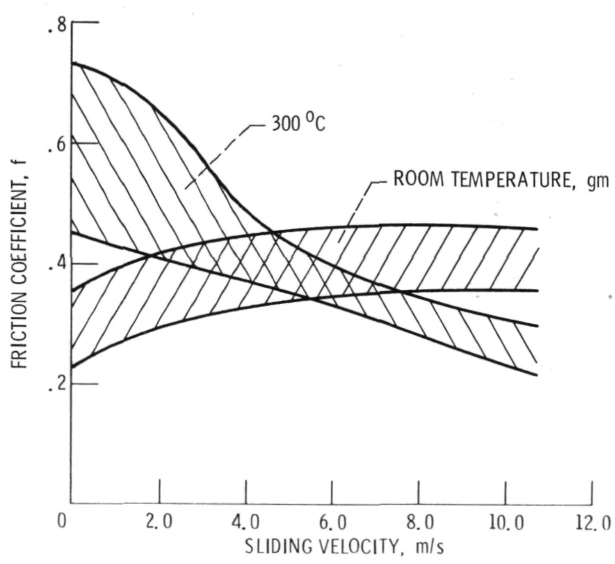

Figure 5. - Alpha SiC sliding on M50 steel at room temperature and at 300 °C in air-1 to 20 N load.

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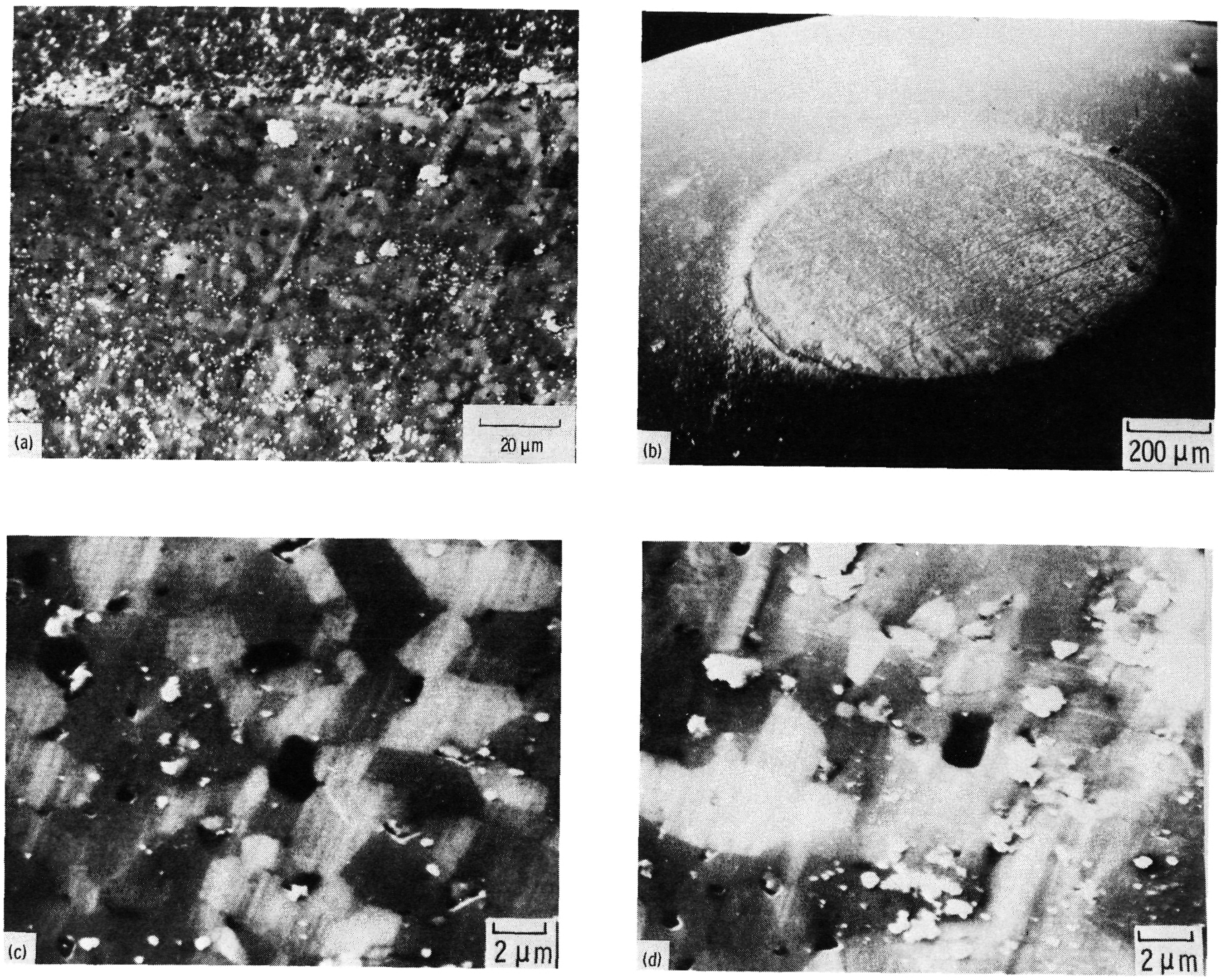

Figure 6. - Alpha SiC after sliding on M-50 steel in air at 300 ^{0}C.

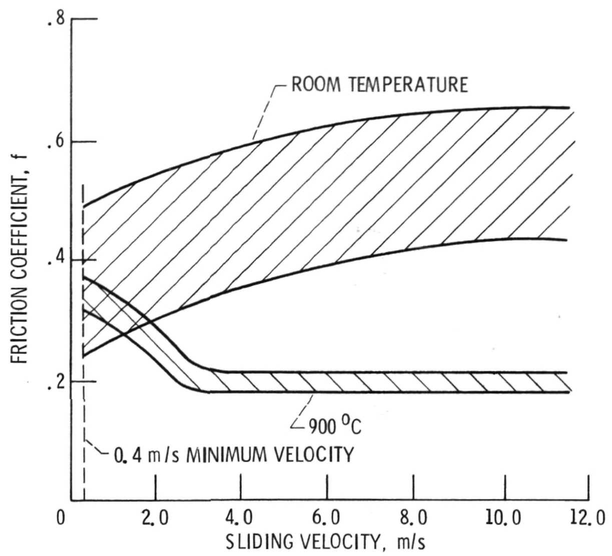

Figure 7. - Alpha SiC sliding on Inconel X-750 at room temperature and 900° C in air, 1 to 20 N load.

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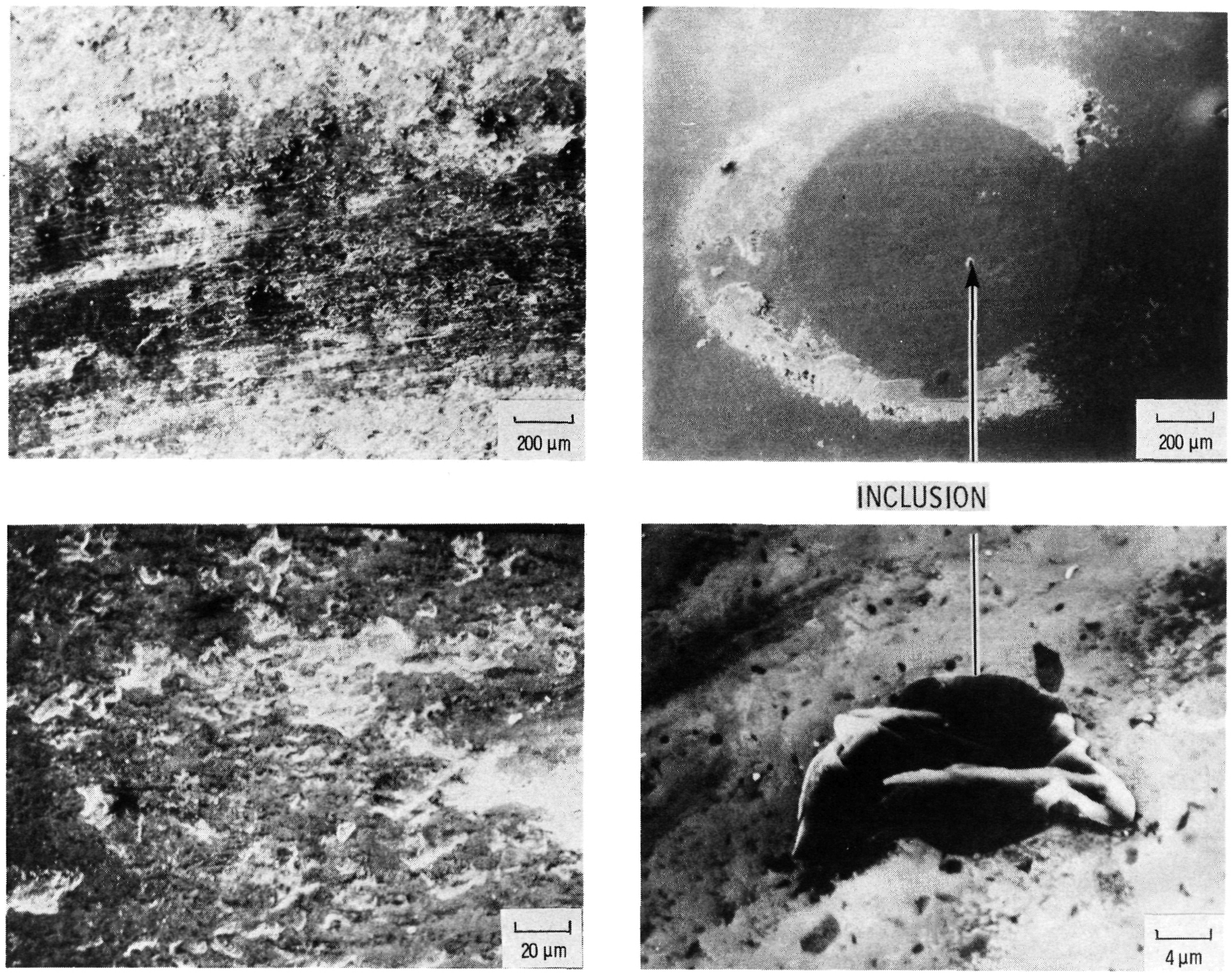

WEAR TRACK SEGMENT ON INCO X-750 DISK.

WEAR SCAR ON α-SiC PIN.

Figure 8. - Sliding contact areas on INCO X-750 and alpha SiC 900 °C in air.

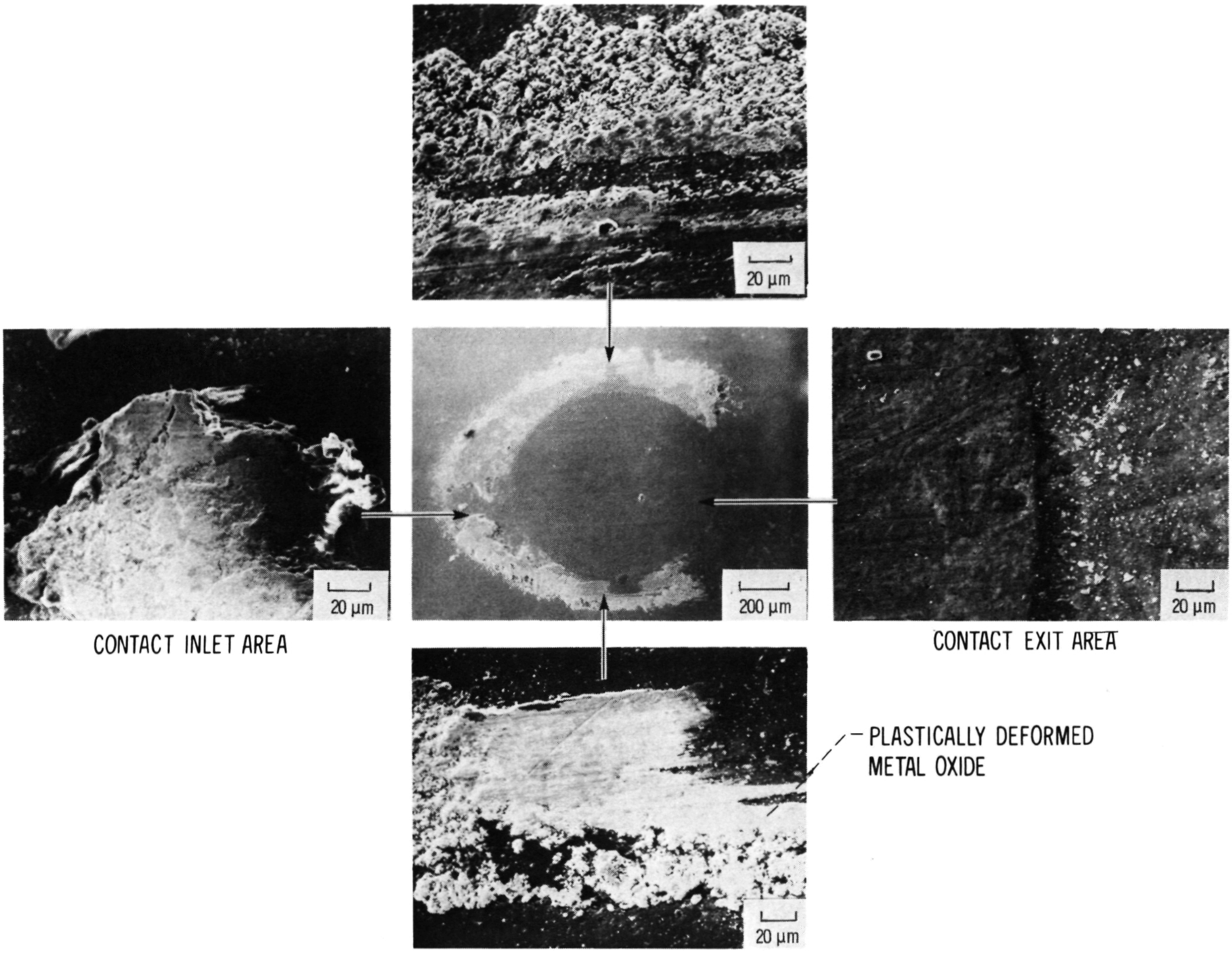

Figure 9. - Alpha SiC pin after sliding on INCO X-750 in air at 900 °C.

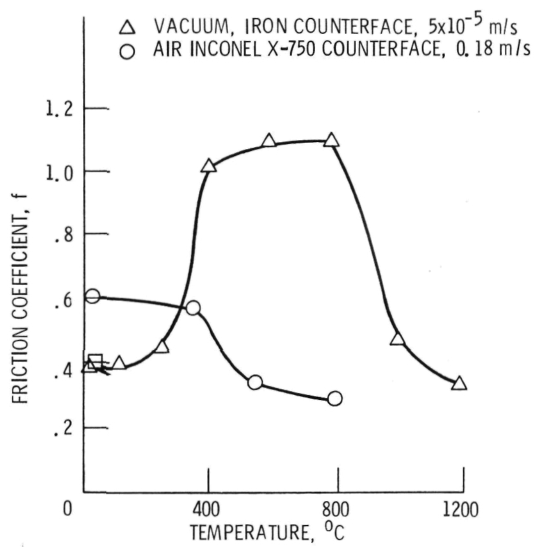

Figure 10. - Effect of temperature on friction of Alpha SiC on metals in vacuum and in air.

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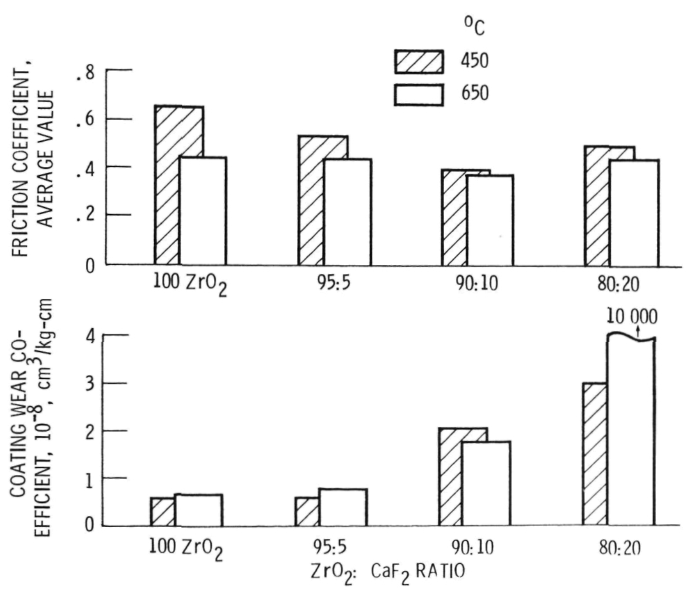

Figure 11. - Effect of CaF_2 additions on friction and wear of ZrO_2 plasma sprayed coatings.

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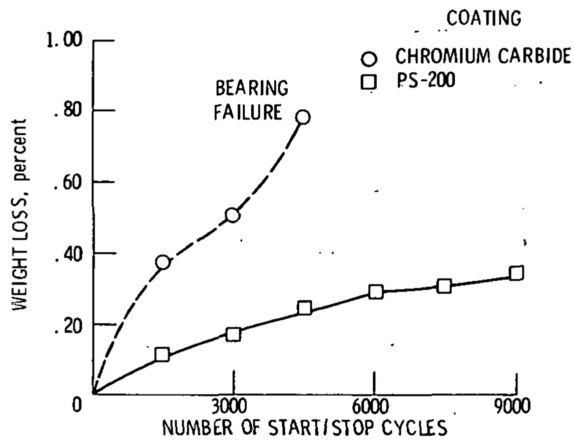

Figure 12. - Comparisons of wear profiles of preoxidized Inconel X-750 foil bearing as run against plasma sprayed chromium carbide and PS-200 coated journals.

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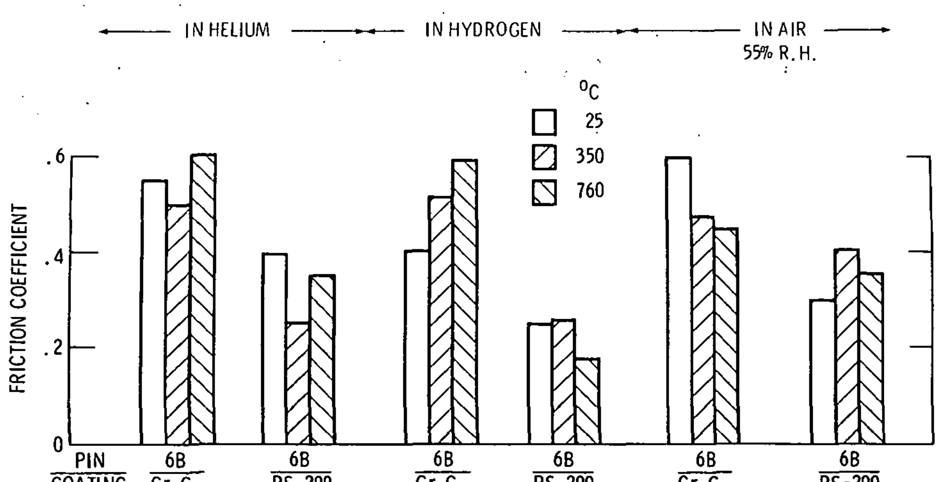

Figure 13. - Friction of Stellite 6B sliding on bonded chromium carbide and on PS-200, 2.7 m/s, 5N.

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16. Abstract

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